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ROLE OF INTERFACES AND INTERPHASES IN THE EVOLUTION MECHANICS OF MATERIAL SYSTEMS

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This is the first "annual" report, covering twelve months of the initial effort on this program. Progress is reported in the characterization of interfaces, the correlation of interfacial quality and global properties, the development of tests for interfacial effects, and the mechanics analysis of microstresses including interfacial effects. A salient result is the achievement of the first solution of interfacial stresses in materials which have locally nonuniform properties.

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Summary

The general objective of this investigation is to apply the discipline of mechanics to the prediction and description of the long-term behavior of composite materials by developing experimental information, conceptual understanding, and analytical representations of the evolution of the properties of constituent materials and interfaces in composite material systems as a function of (generalized) time during the application of time-variable mechanical, thermal, and chemical loadings. The general approach to this objective is to develop mechanistic representations of the "state of the material" under those conditions, and to join those descriptions with micromechanical descriptions of the "state of stress" in "critical elements" to support an estimate of remaining strength, and, thereby, to predict remaining life. We have named this enterprise "evolution mechanics."

The current program focuses on a particular aspect of this general objective, the study of the "Role of Interfaces and Interphases in the Evolution Mechanics of Material Systems." This Annual Report for 1989 summarizes some of the activities conducted under this grant during the first eleven months of the program. The planned activities of the program for the first three years are as follows.

First Year of Investigation:

Material System-

The program will begin with the use of Graphite/Polysulfone composite material, made by commercial suppliers or by the processing group at Virginia Tech. The fiber-matrix interface for that material system is known to be strong under proper processing conditions.

Experimental Program-

The experimental program for the first year will begin with an effort to learn how to make Gr/Polysulfone composite material which has a strong fiber-matrix interface, and how to make the same material with a very weak interface for control experiments. Second, a strong effort will be made to establish nondestructive methods of determining the strength or relative strength of interfaces between the fiber and matrix. The ultrasonic microscope, other ultrasonic methods, electronic methods, and thermal methods will be evaluated for use. Third, using the model materials, test methods for compression, tension, and shear strength measurement will be established, and the effect of interfacial strength on the quasi-static properties at the global level will be determined.

Analysis-

The code NDSANDS will be obtained, and the possibility of altering the unit cell to allow interface and interphase variations will be investigated with the code developers (N. Pagano & associates). Micromechanical and mechanics of materials formulations approaches for tensile, compression, and shear which include the interface/interphase influence will be investigated or postulated.

Second Year of Program:

Material System-

During the second year of the program a composite system known to have relatively poor interfacial strength will be used, possibly one of the newer thermoplastic matrix materials such as Ultem, etc.

Experimental Program-

An attempt will be made to obtain or develop a composite material system with intermediate fiber-matrix interface strength, and to develop NDE information that quantifies that strength in-situ. The weak, strong, and intermediate interface/interphase materials will be subjected to long-term mechanical and thermal loading to determine the degradation of global strength under creep and fatigue conditions. The degradation of the fiber-matrix interface will be correlated with the global changes. Localization of damage due to interactions across interfaces and interphases will be studied.

Analysis-

Using the analysis methods investigated in the first year, stress states in the interface/interphase region of a representative volume of material will be described for tensile, compression, and shear loading.

Third Year of Program:

Material System-

Based on the experience of the first three years, we will attempt to design a composite material system with a specific strength, probably an improved strength for one of the prior systems considered. Other material systems may also be considered. Ceramic/ceramic systems may be used, or polymer-matrix materials synthesized here at Virginia Tech may be considered.

Experimental Program-

The experimental program will focus on the application of the experience and expertise developed to this point to investigate the concept of "toughness", especially as it is related to fiber-matrix interface, especially under long-term loading of specific material systems.

Analysis-

The analysis capabilities developed will be combined with the material characterizations and representations (especially the viscoelastic and damage-related constitutive representations) to describe critical element behavior in a performance simulation code such as MRLife1 developed by the Materials Response Group. This mechanistic representation will replace phenomenological representations such as the SN curve, combine fatigue and creep effects (for the first time), and bring the first rigorous representations of fiber-matrix interface properties and behavior to the composites technical community.

In the remainder of this section, we will summarize the progress made in the first seven months on this program, using the headings listed above. The following section will present selected results from the program activities to date.

Outline of 1989 Program Accomplishments

Literature Search

The first activity undertaken was an extensive literature search and a corresponding analysis of that literature. The results were quite surprising. While the chemistry of interfaces and interphases is widely discussed, and mechanics treatments of "interfacial stress" are available in the literature, the mechanics of this subject is generally undefined, poorly set, and not representative of the actual physics involved. A few points are listed to illustrate this point.

- In many modern composite material systems, especially the thermoplastic matrix materials of great importance to recent applications, the matrix properties are not constant from point to point; in particular, the morphology of the matrix is influenced in a very orderly and significant way by the presence of the fibers. This nonuniform property problem is wholely ignored in the literature (although one author at least attempted - and failed - to address this problem correctly).
- While interfaces are considered, mechanics representations of the interphase (a region of finite thickness) is rarely considered, and that problem is very poorly addressed for the transient stress case near the end of broken or short fibers where the interface or interphase has the most influence on physical behavior.
- There is great confusion associated with the relationships between interface/phase properties and composite properties. The maximum strength interface/phase does not give the maximum strength composite, for example, but a clear description of that problem has not been presented, for example.
- Most of the rigorous and correct models of the mechanics of interface/phase effects (including those of Pagano, Budiansky, and others) are "dilute solution" formulations; the interaction of the fibers with one another is not present in those representations. This is the source of one of the greatest misconceptions currently present in the literature, that the "ineffective length" controls the tensile strength of composites. In point of fact, the local stress concentration in neighboring fibers controls that strength for many (perhaps all) of the composites in current use.
- The influence of the degradation of interface/phase properties during long-term service on composite properties and performance has not been addressed.
- The influence of the variation of the thickness of the interface/phase and the relative thickness of the interface/phase to that of the matrix region and fiber dimensions on composite properties and performance has not been systematically studied or analyzed.

Many other shortcomings and opportunities were identified. A paper discussing some of the conclusions was presented at the Fall meeting of the American Society for Composites, held at Virginia Tech in October of 1989.

Material System

The materials to be used in the present program will be almost entirely thermoplastic matrix composites, mostly with graphite reinforcement. In addition to the practical importance of such material, this choice was made to benefit from the concentration on that class of materials at Virginia Tech in the Virginia Institute for Material Systems and the NSF Center for High Performance Polymeric Adhesives and Composites. Much progress has been made in the acquisition of materials.

1. After visits and discussions with the Cyanamid Corporation, they have agreed to supply composite panels made using their 7005 resin system reinforced with three different fibers which bond "well, not so well, and poorly" with that matrix, based on their experience. The first ten lb. of that material has arrived and will be tested shortly.
2. ICI has provided prepreg material with weak, strong, and intermediate strength interfaces, again, according to their experience. That material differs only in the fiber type and interface treatment. This will provide another essential comparison.
3. Under funding from the Virginia Institute for Material Systems, other investigators at Virginia Tech have made prepreg and panels of thermoplastic composites from "scratch," starting with fibers and resin. That group has also obtained extensive equipment to treat the surface of the fibers, including a plasma treatment facility. This capability, believed to be unique in the United States as a university facility, is available to this program; we intend to use it to make our own weak-strong interface materials for systematic controlled testing and analysis in the second year of the program.
4. We have designed a model material system, and have purchased constituents for that work. The first samples have been made, and one has been tested. Several difficulties with technical matters such as the effects of gripping and the thermal residual stresses are yet to be resolved. However, the stress fields around fibers (including broken fibers) can be studied with this material system.

Obtaining material for interface studies is a very great challenge. We have had reasonable success in getting controlled materials so far. This will greatly facilitate our studies.

Experimental Program

Among the highlights of the experimental effort are the following activities.

1. Material has been obtained and a test matrix has been planned to provide data on the relationship of interfacial properties and characteristics to composite mechanical properties and performance. Because of the extreme difficulty associated with obtaining material which has measureable differences and quantifyable preparation, this part of the program is being conducted with extreme care. With the material available, quasi-static tests, fatigue tests, and some compression strength after impact tests will be conducted. More tests will be planned as material is available. A more complete discussion of this topic appears in the next section.

2. A method of degrading the interface in-situ to examine the effect of that degradation on short- and long-term properties and performance has been conceived and designed. Work will start shortly on the apparatus for that part of the effort.
3. Several methods of characterizing the interface have been examined, and several are being studied closely. These include:
   - Dynamic Mechanical Thermal Analysis (DTMA)
   - Acoustic microscopy
   - Dielectric Analysis (DEA)
   - Nano-indentation in an SEM
   - Micro-indentation using a microhardness tester
   - Stress Pattern Analysis from Thermal Emission (SPATE)
   - Acoustic Emission

   Additional chemical characterization of the interfaces will be available from the other investigations mentioned ear'icr that parallel this program. Surprisingly positive results have been obtained so far with the DMTA method. Also, a new initiative to use and develop the analysis to interpret the results of micro-indentation with a standard microhardness tester is under way. Initial results are very encouraging. This technique may present a new method for the characterization of the shear-related properties of interfaces in-situ.
4. A model material has been conceived and designed. Materials have been purchased and attempts are being made to develop a method to manufacture these systems. The model uses 0.125 in. glass/epoxy rods imbedded into a photoelastic matrix material (epoxy is currently being tried). We hope to use this system to isolate the ***mechanical*** part of the interface/phase influence on the strength and life of composites. We especially hope to study the local stress concentration at fiber breaks as a function of the interface.
5. With assistance from Virginia Tech and the Virginia Institute for Material Systems, we have established a new "Interface Mechanics Laboratory." That laboratory has a new work surfaces and cabinets, a new bench metallograph, a new microhardness tester, new cutting and polishing equipment, and a variety of photography devices. Other additions are planned. This is a major addition to our capabilities in this area, and one of the few laboratories in the country to concentrate on the ***mechanics*** of interfaces. (It may be the only one.)

Analysis

Major progress has been made in the development of analysis to properly represent the mechanics associated with interfaces and interphases. The activities include the following:

1. The NDSANDS program has been obtained and throughly investigated. The investigators (a group of six faculty and students) traveled to Dayton Ohio by van and spent the day with the developers of the code (Pagano, et.al.) at the Adtech Corporation and at Wright Patterson AFB. A review of interface mechanics activities was presented with presentations from the Virginia Tech group and the Dayton group of investigators. The NDSANDS code was demonstrated, and copies were provided for the Virginia Tech research team. This is the first copy of that code to be used by a university. The code was used for a thorough "shakedown study"

which concentrated on the influence of interface properties and geometric proportions. Details of that study will be presented at a later time.

2. A thorough study of the literature was conducted to determine the "state of the art" in the analysis of interface/phase stresses and strength. The most recent four-phase model developed by Pagano and Tanden, which is the basis for the NDSANDS code, was completely derived, including all algebra. Shortcomings and limitations were identified.
3. Several formulations are under way to make the "next step" in the mechanics analysis of interphases. The primary focus of this work is the addition of the capability to incorporate the spatial variations of properties (mentioned earlier) known to occur in many modern composite material systems, and to eliminate the "dilute solution" assumptions which so badly distort the conclusions of such current analysis. Some early success has been obtained, and will be presented at a later date. This activity is well under way.
4. A recent analysis by Whitney and Drazel has been studied. The expressions were "rederived" and checked, along with the assumptions and limitations associated with that approach. That analysis is particularly interesting because of the relative simplicity of the approach, and the possibility that it can be altered to account for fiber-fiber interactions and used to calculate accurate local field stress concentrations. The analysis does not have an interface/phase representation as published; other approximations and limitations have discouraged us from further use of that approach.

At present, we have state-of-the-art capability to analyze the mechanics of interfaces and interphases, including the effects of temperature, moisture, impurity phases, and constituent properties, as well as the dimensions of the inter-region (as noted above). We are well along in the development of the next generation of analysis that will remove the serious limitations that we have identified in our study of the existing models and approaches.

All elements of the program are well under way, and most are on schedule or ahead of schedule. The acquisition of specially prepared material, the development of new laboratory facilities, and the development of new analytical capabilities will provide an excellent foundation for the second and third year of the proposed program. This progress will be fully exploited in those parts of the effort, as described in the outline presented earlier. Special attention will be given to the establishment of baseline properties and performance in the remainder of this year of work, and to the completion of the analytical formulations that properly represent the interface mechanics that is associated with the "real" physics of the problem. The development of a model material will be completed during this year, and fundamental studies will be conducted in years two and three, especially studies to identify the effect of "mechanical bonding" of the fiber surfaces to the matrix, and studies of the stress concentration in adjacent fibers when a fiber fractures. And finally, micromechanics models will be adapted to the problem of the degradation of interfaces, which will be examined in the laboratory under cyclic loading. These models, based on the analyses under development, will be used in performance simulation codes such as the MRLife series developed in our laboratory, to represent the "Evolution Mechanics" of interfaces and interphases, the primary objective of our research.

In the following sections, three summaries of specific activities will be presented, in sections titled "Review of Progress on the Experimental Program," "Microhardness Testing of Composite Materials," and "Micro-Stresses in a Composite with Elastic Modulus Gradients."

Review of Progress on the Experimental Program

The summary in the previous section alluded to the progress attained in many aspects of our Experimental Program. This section will detail several of those accomplishments.

At the beginning of the program, we solicited several suppliers for material or prepreg which exhibited differing fiber/matrix interphase characteristics. American Cyanamid supplied us with 10 lb. of composite prepreg using their 7005 resin system. This system represents only one of the three material systems that we are expecting from them. ICI has supplied us with three different material systems each possessing a unique interphasial quality. The three materials are:

- Hercules IM-7 unsized fiber/977-2 resin.
- Amoco T650-42 unsized fiber/977-2 resin.
- Celion G40-700 unsized fiber/977-2 resin.

The appeal of these materials to our research effort is readily apparent. In each material the resin system is the same. The discrepancies that ICI has detected between the three materials' response to impact loading has led their engineers to suspect differences in the interphase. Thus, each fiber system may influence the chemical and/or mechanical bond that occurs at the interphase. Our first goal has been to research and develop destructive or non-destructive tests that may differentiate composite response that may be directly attributed to the interphase.

The first step taken towards this goal was an extensive review of the literature. Many tests exist purporting to measure interphasial quantities. They include:

- Single fiber pull-out
- Critical length of a single fiber
- Transverse tension
- Longitudinal flexure (short beam shear)
- Transverse flexure
- Dynamic Mechanical Analysis (DMA)
- Dielectric Analysis (DEA)
- Microindentation
- Fractography of 0° toughness or quasi-static specimens
- Compression Strength after Impact (ICI may have based there classifications on this test)
- Stress Wave Factor

Several shortcomings have been identified in each of the above tests. A decision was made, however, to pursue DMA and Stress Wave Factor testing as non-destructive methods to interrogate the interphase. A sub-section will be devoted to each of the two test methods and will address the motivation behind employing these techniques and the results obtained.

Stress Wave Factor (SWF)

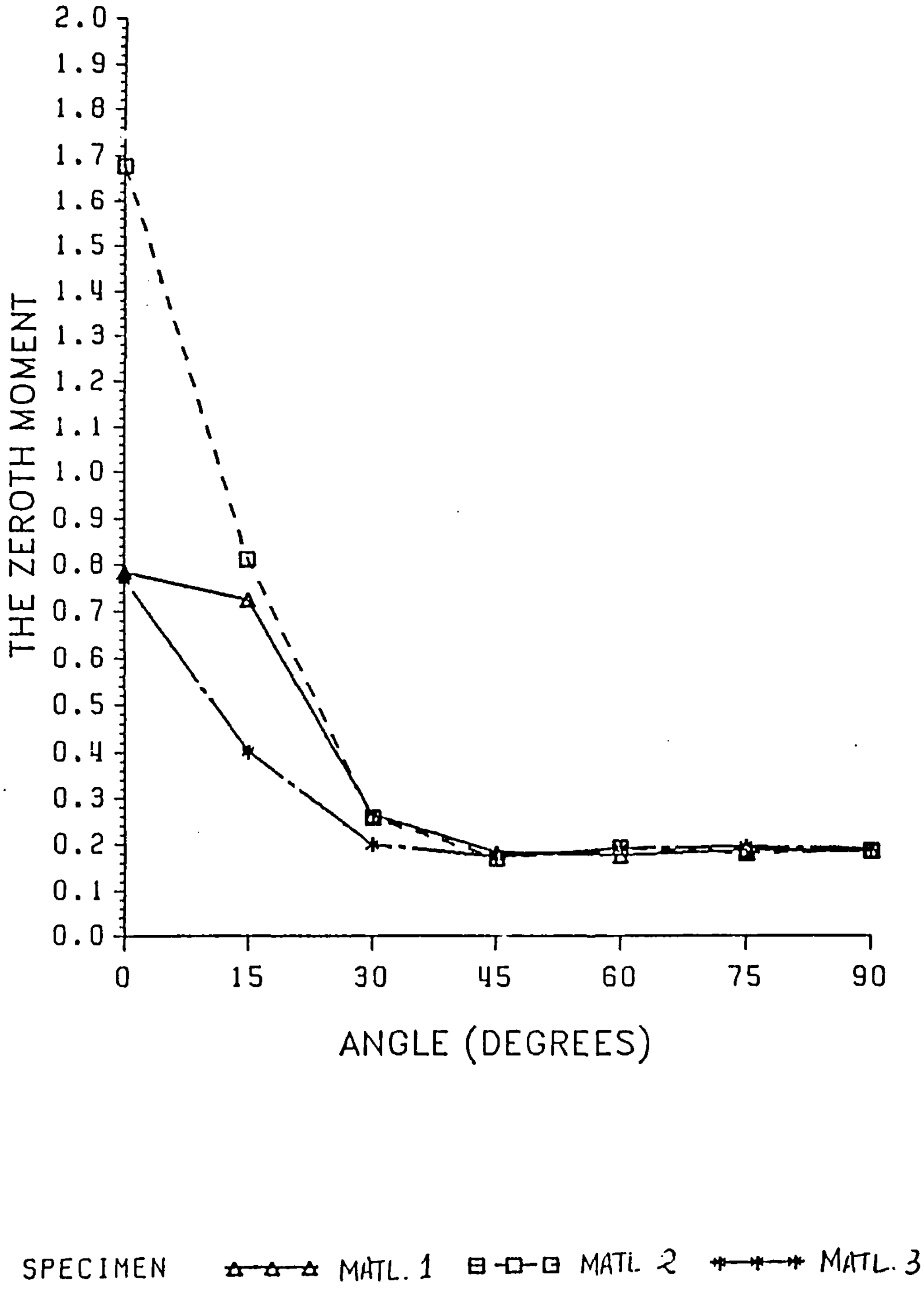

Figure 1. Plot of moment of frequency response about the origin vs. fiber angle for first test.

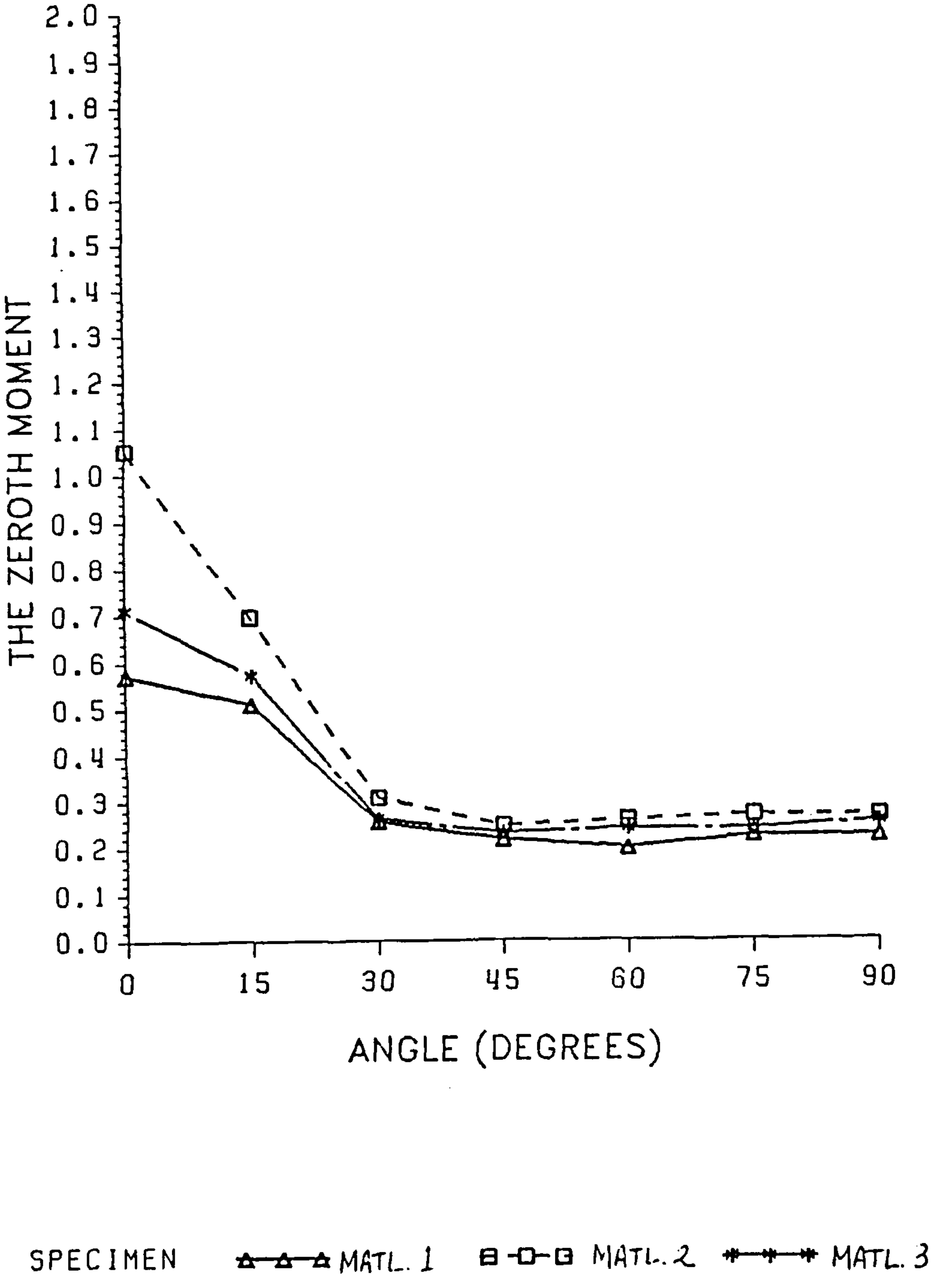

Figure 2. Plot of moment of frequencey response about the origin vs. fiber angle for second test.

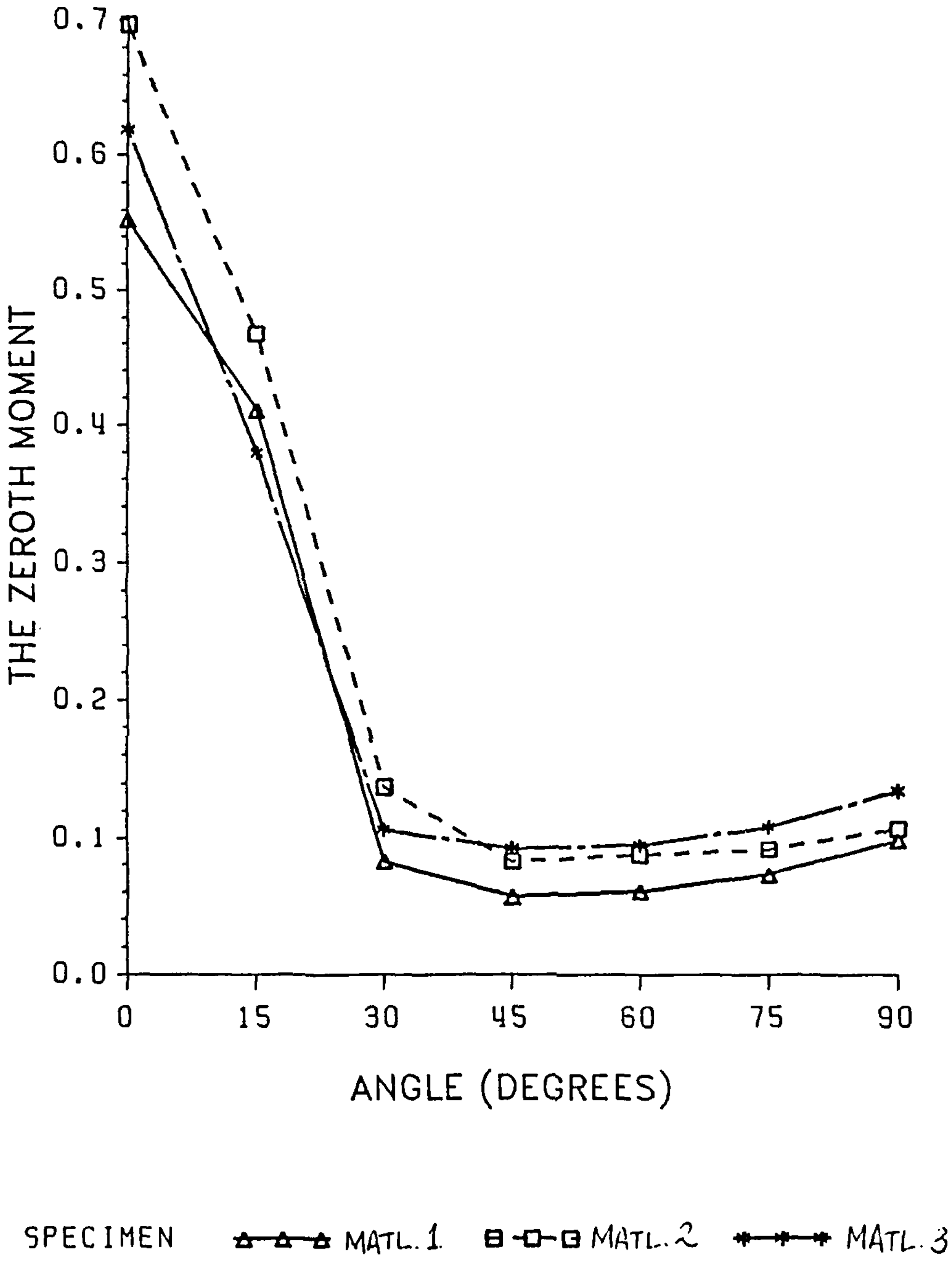

Figure 3. Plot of moment of frequencey response about the origin vs. fiber angle for third test.

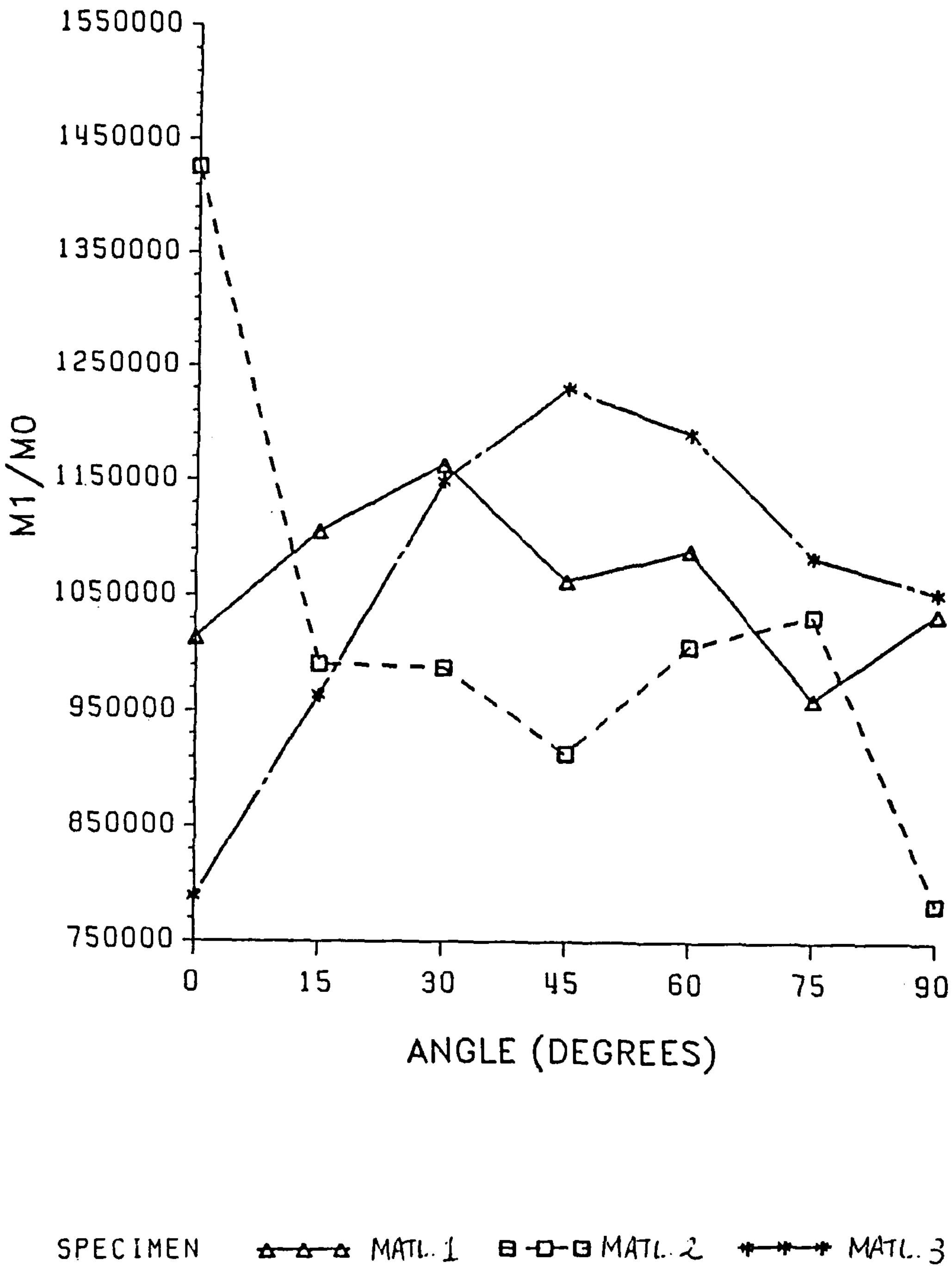

Figure 4. Plot of the ratio of moment of the frequency response about its centroidal axis to the moment about its origin vs. fiber angle for test number one.

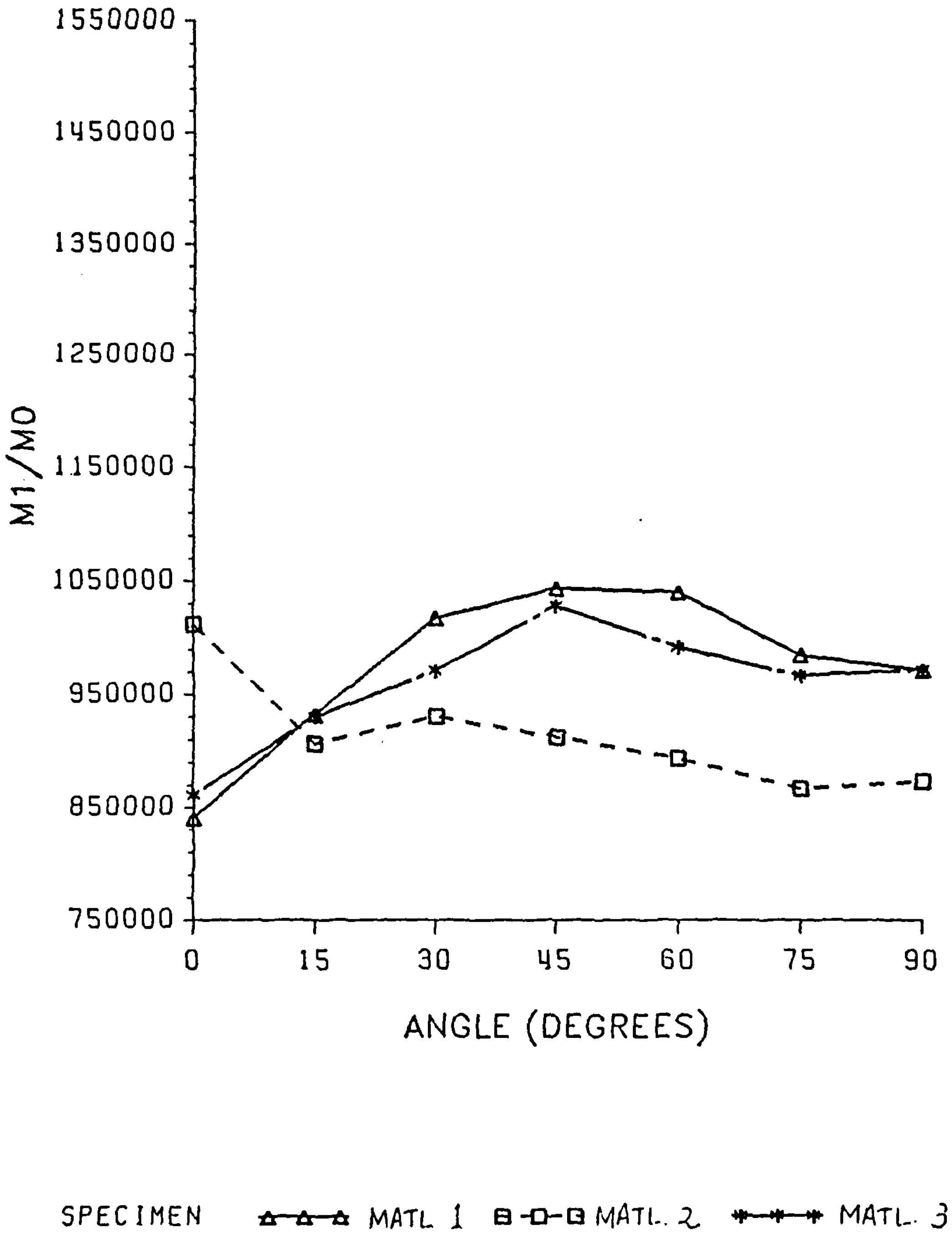

Figure 5. Plot of the ratio of moment of the frequency response about its centroidal axis to the moment about its origin vs. fiber angle for test number two.

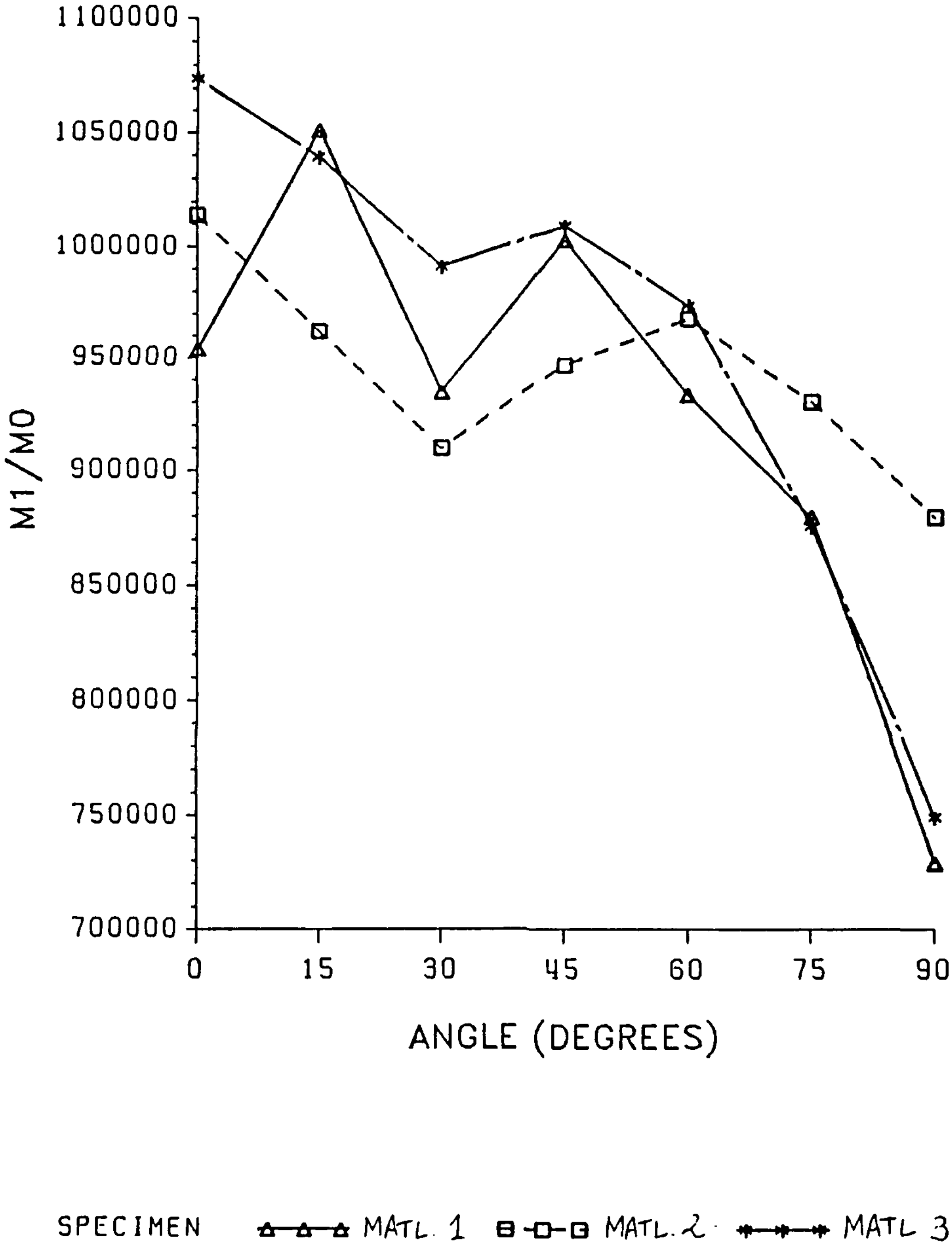

Figure 6. Plot of the ratio of moment of the frequency response about its centroidal axis to the moment about its origin vs. fiber angle for test number three.

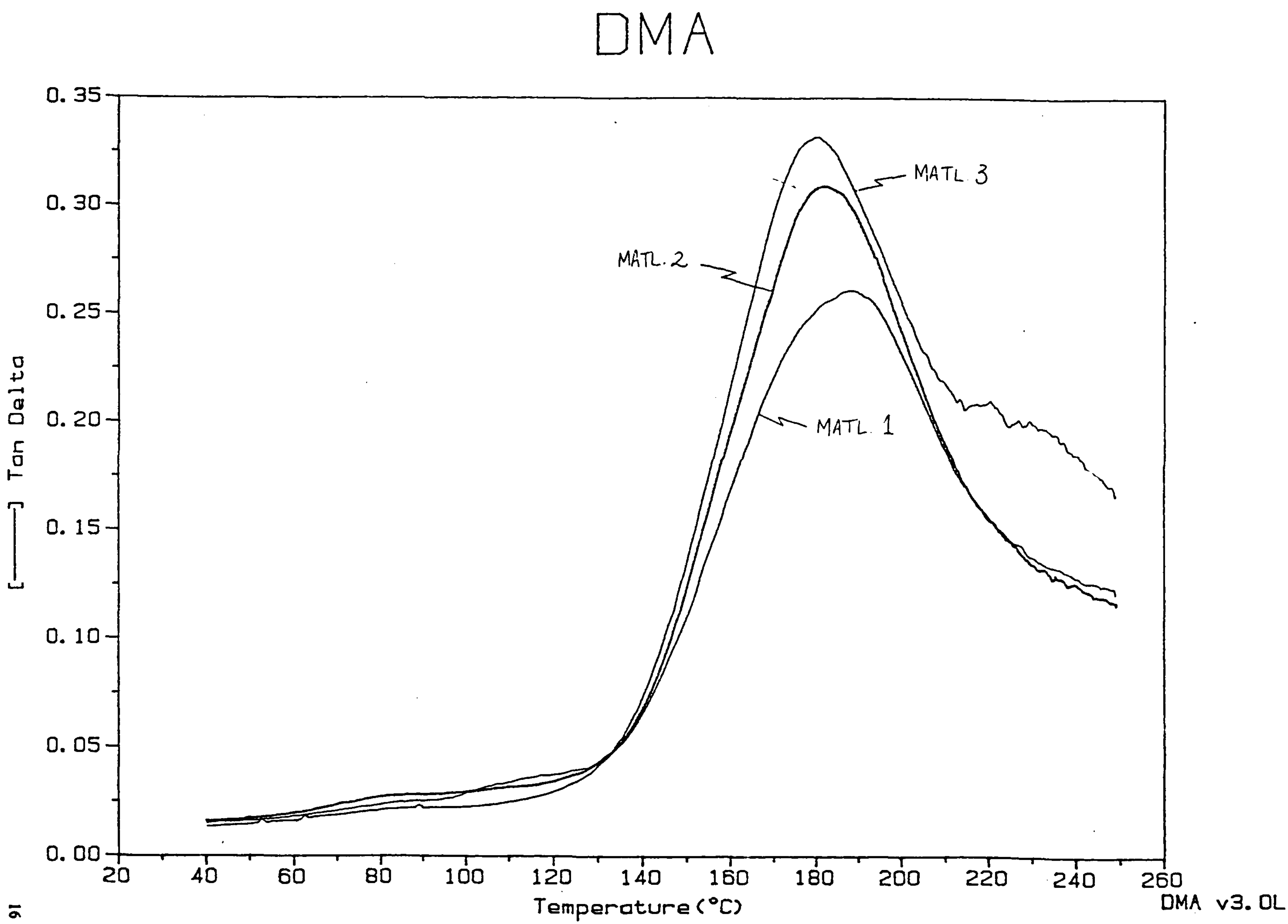

Figure 7. DMA plot of tangent delta vs. temperature for the three ICI materials. All materials are 90° unidirectional and cycled at 1 Hz.

Vary and Lark[1] employed an acousto-ultrasonic (AU) technique in an attempt to measure strength variations in unidirectional composites. They defined a "stress wave factor" and reported that its value depended on "variations of the fiber-resin bonding" Since the publication of this research, Vary, et al., have focused on employing the Stress Wave Factor as a measure of strength variations and has not pursued the use of SWF as a means to qualify fiber/matrix bonding.

Our decision to pursue this method is physically well founded. By imparting a mechanical pulse to the plate, one expects this pulse to be transmitted and, for that matter, dissipated differently depending on the degree of contact between the fiber and the matrix. Intimate contact — a strong fiber/matrix interface — should encourage transmission of stress waves while discouraging dissipation at interfaces. The converse is expected at weak interfaces.

Initial AU studies have been performed on the three different material systems supplied by ICI. Employing the software and hardware described in the paper by Kiernan and Duke,[2] the AU parameters A1 and A2 were plotted versus azimuthal angle for each material. Three separate runs are included (see Figure 1, Figure 2, Figure 3, Figure 4, Figure 5, Figure 6). These early tests indicate that the three materials do, indeed, appear different to the AU inspection. Differences in the three tests can be contributed, at least in part, to differing test equipment parameters (ie., gain, gate length, position of gate, etc.), yet, there is some concern about reproducibility. These results, however, merit further investigation of the utility of the AU technique to our study. We have considerable confidence that this method will reveal to us previously unseen trends in material behavior.

Dynamic Mechanical Analysis (DMA)

Chua[3] has investigated the use of DMA as a measure of interphasial bonding. He reports, "the interfacial shear strength and the tan delta at the glass-transition temperature of the glass-fiber reinforced polyester show good correlation suggesting that the latter can be used to characterize the quality of the interphase." The physics involved in this method are not unlike that of the SWF. The DMA measures the phase lag (delta) that occurs between the applied displacement and the resulting stress. For a perfectly elastic material this phase angle is zero. However, a dissipative material will exhibit a measurable phase lag between the two responses. The following reasoning may be applied to the interphase: If imperfect bonding exists between the two phases then one would expect this to be a source of energy dissipation, especially when compared to a material with perfect bonding. Those materials that comparatively reveal a larger phase lag (proportional to tan delta) are dissipating more energy. Therefore, if those mechanisms that normally cause dissipation

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[1] "Correlation of Fiber Composite Tensile Strength with the Ultrasonic Stress Wave Factor," **Journal of Testing and Evaluation,**, JTEVA, Vol.7, No.4, July, 1979, pp. 185-191.

[2] "Acousto-Ultrasonics as a Monitor of Material Anisotropy", **Materials Evaluation,** July, 1988, pp. 1105-1113.

[3] "Characterization of the Interfacial Adhesion Using Tan Delta", **Polymer Composites,** Vol.8, No.5, October, 1987, pp. 308-313.

(i.e., the viscoelastic matrix) remain constant between different materials, one might attribute the differences in dissipation to the existing bonding condition.

DMA testing was conducted upon the three material systems supplied by ICI. Since the only difference between the materials is the reinforcing fiber, one may be tempted to conclude that differences in the DMA response may be attributed to the interphase. The results of a series of DMA runs are shown in Figure 7. This figure displays tan delta for the three materials as a function of temperature. The three responses have separated into three distinguishable curves (it should be mentioned that variations between separate runs do not deviate significantly from their respective curves). The rationale applied by Chua would conclude that since the phase lag is greatest for the upper peak, this material should possess the poorer interphasial bonding, while the lower peak possesses the best bonding.

The results from the DMA testing have been a positive step in our attempt towards identifying a non-destructive test that qualifies and quantifies interphasial effects on composite performance. The SWF results have unfortunately been clouded with questions of reproducibility. There are plans to correlate these DMA results with other test results, perhaps from tests identified in the literature review. Currently work is being conducted on a microhardness tester in an attempt to differentiate the bonding conditions of the ICI materials (see later section for details). The goal of this work is the emergence of a test method that can reliably measure differences in fiber/matrix bond conditions and/or the effect these differences has on the response of a composite material.

Current work is being performed to satisfy our stated goals for the second year of this program. We have recently identified a process in which to degrade the interphase in-situ. This step will significantly propel our understanding of the role of the interphase on the long-term behavior of composites. The test methods identified for characterization of interphase behavior will be employed to distinguish the response of virgin and degraded interphase composites. The response of degraded composites to typical loading conditions will then be researched in order to extrapolate the role of the interphase in composite performance. Such data will verify the micro- and meso-level models constructed by incorporating not only interphasial properties but the *degradation* of interphasial properties. In the next two years the following questions will be addressed:

- Given that bonding conditions can be differentiated, what aspect of the constituents produces these measurable differences in global behavior? Is it due to differences in the Poisson contraction of the reinforcing fibers? Does it primarily depend on surface chemistry, i.e., wetability, the presence of different functional groups, etc.?
- Given that bonding conditions can be differentiated, how does the bonding condition affect the specimens' long-term performance? Does a "strong" bond prolong cross-ply fatigue life more than an "optimal" bond? Why?
- Does interphasial degradation occur over long-term loading? If the interphase does degrade, how does this affect the performance?
- How does one incorporate an interphase into micromechanical analyses and permit the degradation of the interphase using conventional (and, perhaps, unconventional) mechanics expressions.

Microhardness Testing of Composite Materials

Introduction

The hardness of a material provides a measure of its resistance to permanent deformation. More specifically, hardness pressure has been empirically related to the compressive yield strength for homogeneous and isotropic materials. Present microindentation techniques for composites measure the de-bond strength of a fiber by pushing it from its surrounding matrix. The system makes it possible to obtain repeatable interfacial strength information on commercial materials, and does not require a special specimen configuration. However, data collected is limited to the fibers selected and may not be a representative average of the entire composite. In addition, the surrounding matrix is not loaded and does not produce a realistic stress situation for the composite. Poissons' effects may play a role in the interfacial strength, particularly when the bond strength is weak.

In an attempt to develop an alternate approach for measuring interfacial properties, conventional microhardness techniques have been attempted with two primary goals:

1. Correlate hardness information obtained from conventional tests to engineering properties of composites.

2. Investigate the possibility of isolating interfacial properties from fixed load and continuous indentation techniques.

Findings

The Vickers microhardness test was chosen for initial study due to the unity aspect ratio of the impression. The penetrater is a diamond ground in the shape of a square based pyramid with an angle of 136 degrees between faces, as depicted in Figure 8 on page 20. The Vickers Hardness (HV) number is the quotient of the applied load divided by the sloping area of the plastic impression. Experience has shown that Vickers hardness of homogeneous materials is independent of load, except at very light loads.

Two thermoplastic composite systems were chosen for initial study because of their known relative difference in interfacial strength. AS4/J2 was considered to have a "strong" interface compared to AS4/DuP in which the matrix material does not readily adhere to other surfaces.

Indentations were initially made perpendicular to the fibers (3-direction). The anisotropic nature of the composite caused some difficulty in measuring the diameter of the indentation due to the large amounts of elastic recovery in the fiber direction. Optical microscopy of the damage produced by the penetration revealed significant crack growth along the fiber length in the

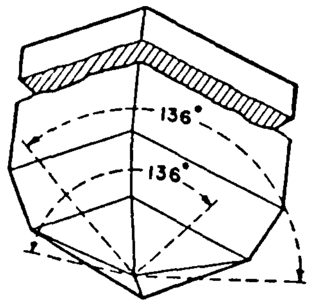

Figure 8. The Vickers diamond indenter.

AS4/DuP. The AS4/J2 displayed much better fiber/matrix stress transfer which was observable in crack growth perpendicular to the fibers. Further, AS4/J2 exhibited a HV of 35 kg/mm^2, more than twice that for the AS4/DuP (HV = 15 kg/mm^2). The primary reasons for this difference is due the relative stiffness of the matrix (J2 is about 2 times stiffer) and volume fraction (AS4/J2 v_f = 0.6 and AS4/DuP v_f = 0.4).

Penetrations in the fiber direction (1-direction) were considered to be a better indicator of the composite properties. In addition, the symmetry in this plane made it easier to distinguish the plastic indentation, which allowed for precise computation of HV. Specimens were mounted in plastic and polished to a metallographic finish. The ends of the fiber and surrounding matrix were clearly distinguishable. Indentations at loads ranging from 20 to 1200 grams were each made on undamaged regions of similar fiber distribution. Indentation diameters (diagonals of the plastic perimeter) ranged from 330 to 15 microns. Typically, the penetrater contacted a representative region of both fiber and matrix (fiber diameters are about 5 microns).

The results of plastic penetration depth versus load is shown in Figure 9, and clearly indicates that AS4/J2 has a higher resistance to penetration. Examining the same data on a log-log scale, shown in Figure 10, reveals a small transition, represented by a change in slope, signaling different modes of deformation. Further study is needed to define the phenomenon which accompany these findings. Investigating how hardness varies with load may supply some of this information. For these composites the hardness does not remain constant even at higher loads, as depicted in Figure 11. The sharp increase in hardness at low loads is due to the elastic recovery of the true indentation diameter upon removal of the load. The proportional reduction in plastic contact area increases the apparent HV, given the percentage of elastic recovery. (Ideally,

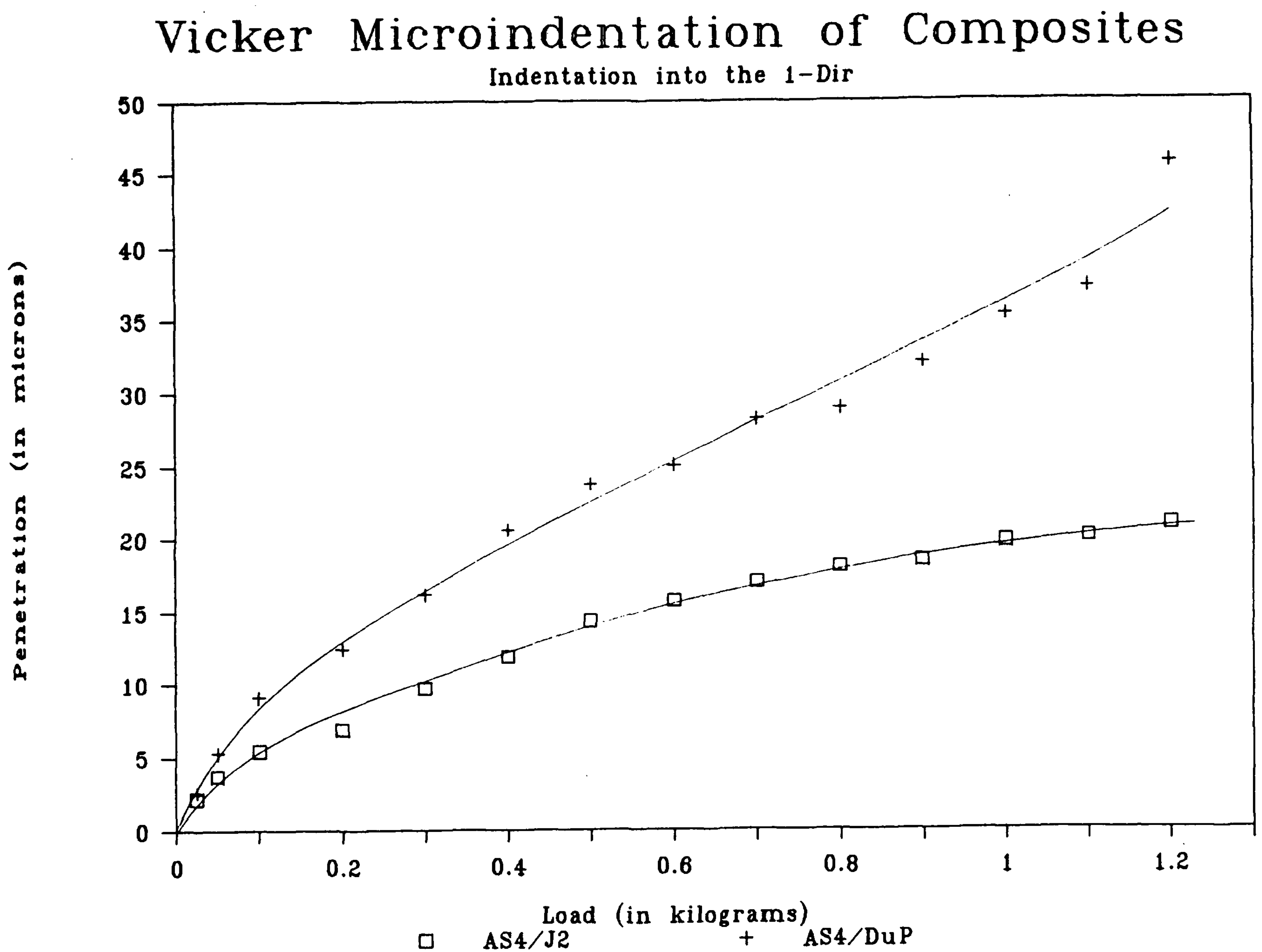

Figure 9. The plastic penetration depth versus load from the fixed load microindentation of AS4/J2 and AS4/DuP.

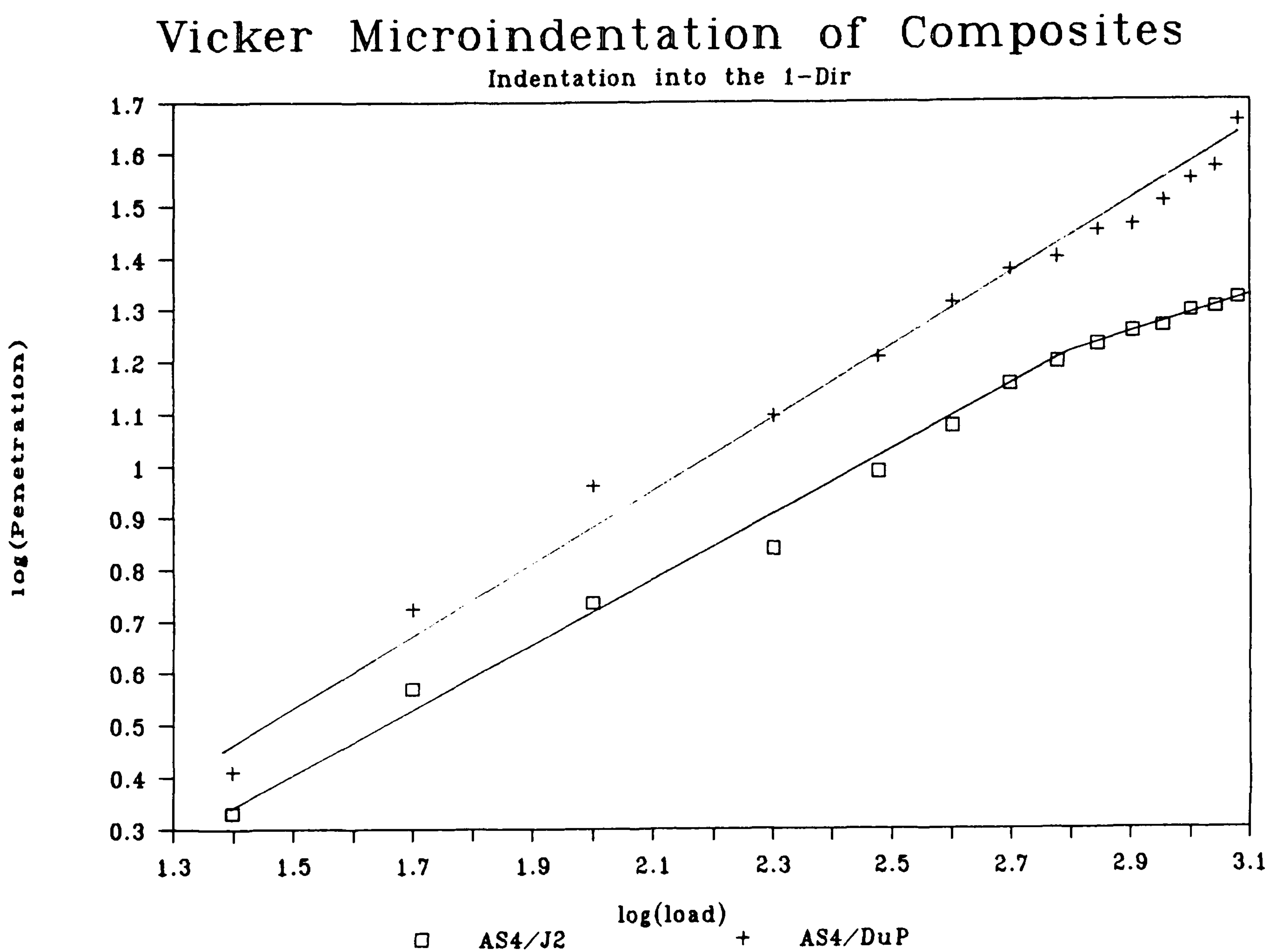

Figure 10. The plastic penetration depth versus load (log-log) from the fixed load microindentation of AS4/J2 and AS4/DuP.

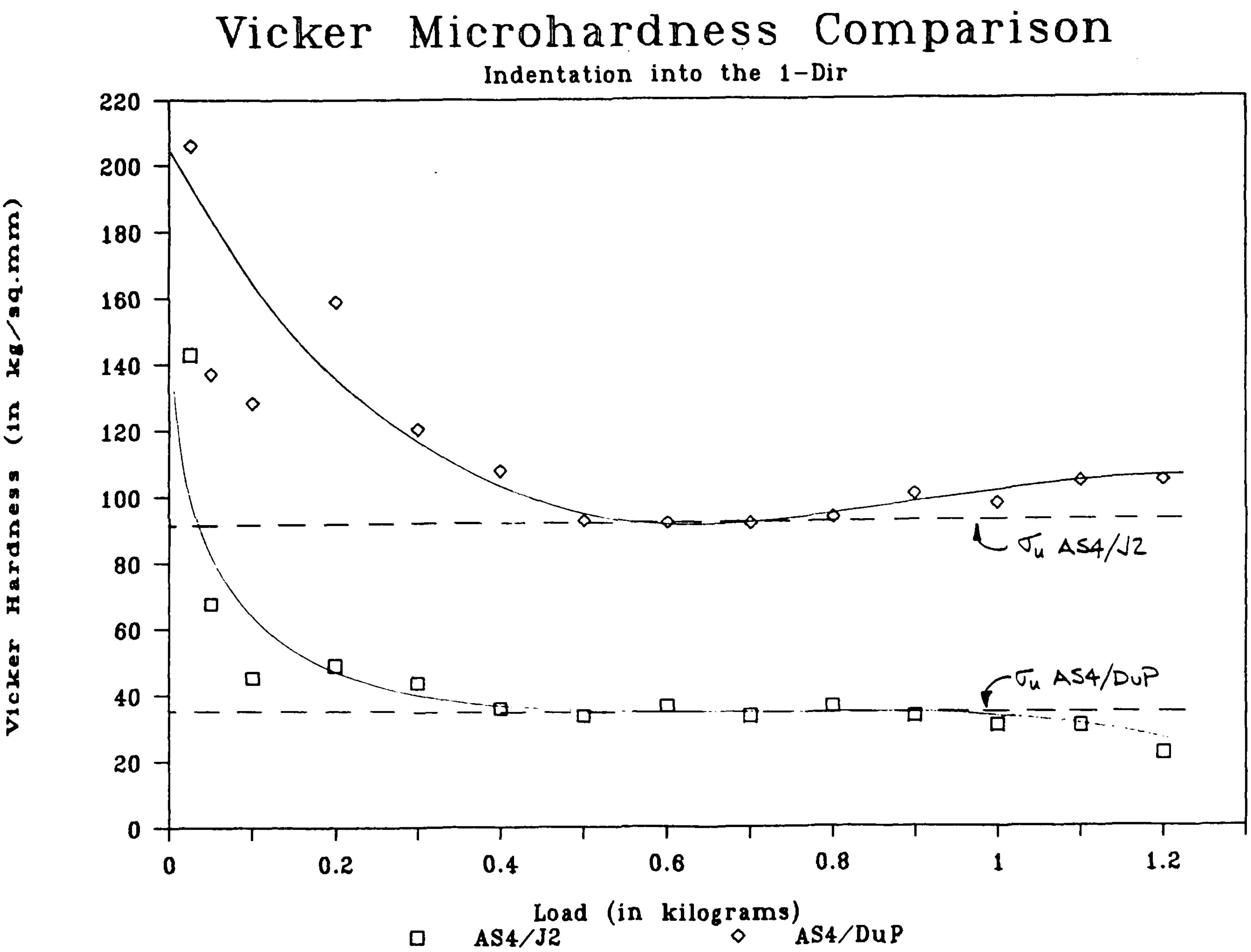

Figure 11. The Vickers hardness versus load for the fixed load microindentation of AS4/J2 and AS4/DuP.

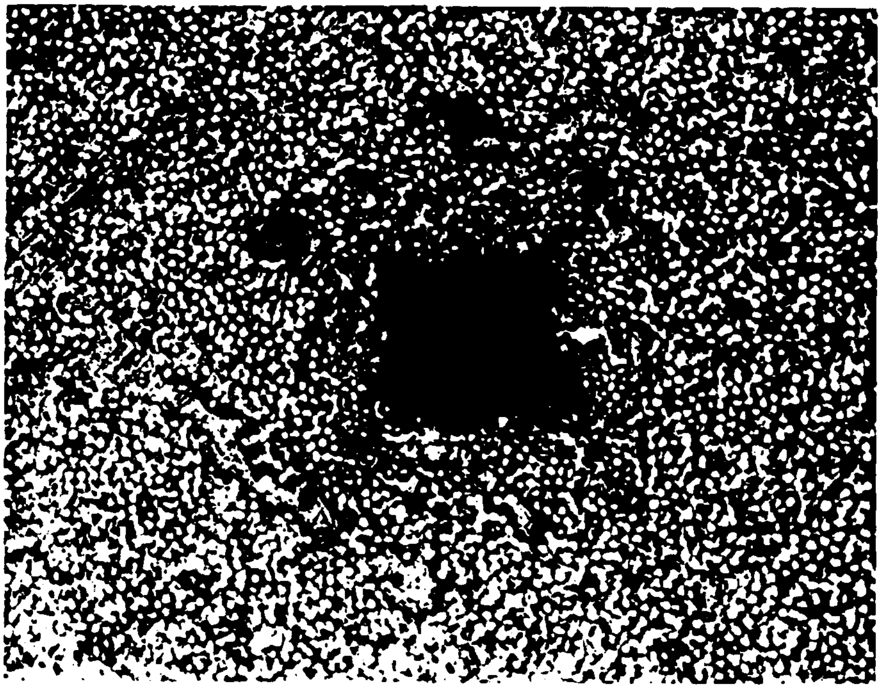

Figure 12. A photomicrograph of a Vickers 1200 gram penetration into AS4/DuP. The plastic diameter is about 320 microns across. The up-lifted matrix is seen as the blurred regions around the residual impression.

below the elastic limit the HV would be infinite.) Note the greater percentage of elastic recovery for the AS4/J2.

In addition to the hardness testing, unidirectional 0 degree compression tests of the two materials were completed in accord with the ASTM standard D-3410. The ultimate compression strengths achieved are close to the HV values found for fixed loads above 0.5 kg. These results are represented as horizontal lines in Figure 11. The agreement was very close, suggesting the possible utility of this technique. If further correlation is seen on other systems, microhardness testing could be conducted on very small samples, possibly replacing the need for complex and expensive compression tests.

Further observations made with an optical microscope and SEM reveals regions of matrix which are raised up from between the fibers. These zones, not in contact with the indenter, appear as far as 1.3 diameters from the impression center. This phenomenon is displayed in Figure 12were the blurred regions are the up-lifted matrix. The extent of this up-lifted matrix was observed to be greatest in the AS4/DuP. Also, SEM studies confirm the failure of the fiber/matrix interface at similar points and other areas close to the contact region.

Conclusions

At this point no comprehensive conclusions may be made about the role of hardness testing in composite materials. However, the present findings show some promise in the use of hardness testing in composites. The development of such tests would provide an economical and simple means for evaluating small amounts of material.

The following observation are offered:

- The hardness of composites is influenced by matrix stiffness, volume fraction, and indentation direction relative to fiber direction.
- Indentations made in the 1-direction clearly show the failure of the fiber/matrix and plasticity of the matrix material by stresses not induced by direct contact.
- A relationship between ultimate compression strength and hardness is present at particular loads.
- The results of fixed load hardness testing shows trends which may prove valuable in the determination of interfacial strength.

Future Work

- A comparative study will be undertaken of composites with differing fiber systems in the same matrix material.
- A model is sought which will describe the distribution of stress about the effected region of indentation.
- Continuous indentations will be completed to evaluate the combined elastic and plastic responses during penetration.
- Additional techniques in viewing damage caused by indentation will be attempted so as to define the effected zone about the impression.
- Indentations on neat resin and fiber reinforced composite samples will be conducted at several temperatures and loading rates in order to ascertain if there are different activation energies for the two types of specimens. If so, the viscoelastic nature of the interface can be probed.

Micro-stresses in a Composite

With Elastic Modulus Gradients in the Fiber-Matrix Interphase

Abstract

A solution method for a new class of problems essential to the understanding of fiber-matrix-composite behavior has been found and demonstrated. The method has been used to determine micro-stresses in a composite with elastic modulus gradients in the fiber/matrix interphase. Three different non-linear variations were chosen to simulate the elastic modulus in the interphase region around the fibers. The resulting governing field equations are solved directly in closed-form. A parametric study is performed to assess the effects of interphase thickness and fiber volume fraction on the local thermal stress state. Results indicate that modifying the elastic modulus of the interphase as a function of position can have a significant effect on the local thermal stess state.

Discussion

It is widely known that the material properties in many composite material systems are not constant as a function of position. This is especially true in the matrix and the interface/interphase between the matrix and the fiber for polymeric composites, but also true for metal matrix materials and for ceramic composites, especially at high temperatures. However, finding the local stresses in the fiber-interphase-matrix region with spatially variable properties is a very difficult task, one which has not been successfully undertaken up to this point.

Indeed, for such a problem, the fundamental mathematical form of the governing differential equations of mechanics changes with the type of spatial non-uniformity which occurs, resulting in distinctly different solutions (and requiring distinctly different solution schemes) for each case.

This barrier to the correct representation of this essential problem has recently been removed by the discovery of what is thought to be a general approach to these problems. Several such solutions have been found using this approach to date by the present investigators, opening the door to a major advance in this area. A more complete description of the details of this progress will be given in a subsequent report. In the present report, a few salient results will be presented without an explanation of the analysis method, to provide a general indication of the consequences of the work and the nature of the results.

A continuous fiber unidirectional composite is modeled by three concentric cyclinders for the thermal loading problem. It is assumed that the model consists of a fiber (transversely isotropic or isotropic with uniform properties), and interphase (isotropic with non-uniform properties), and the matrix material (isotripic with uniform properties). The properties of T300/Ni/Al 6061 and E-glass/IMHS epoxy were used for the examples shown. Within the interphase regions, three types of variations were considered:

- A power law variation of the form Pr^{Q}

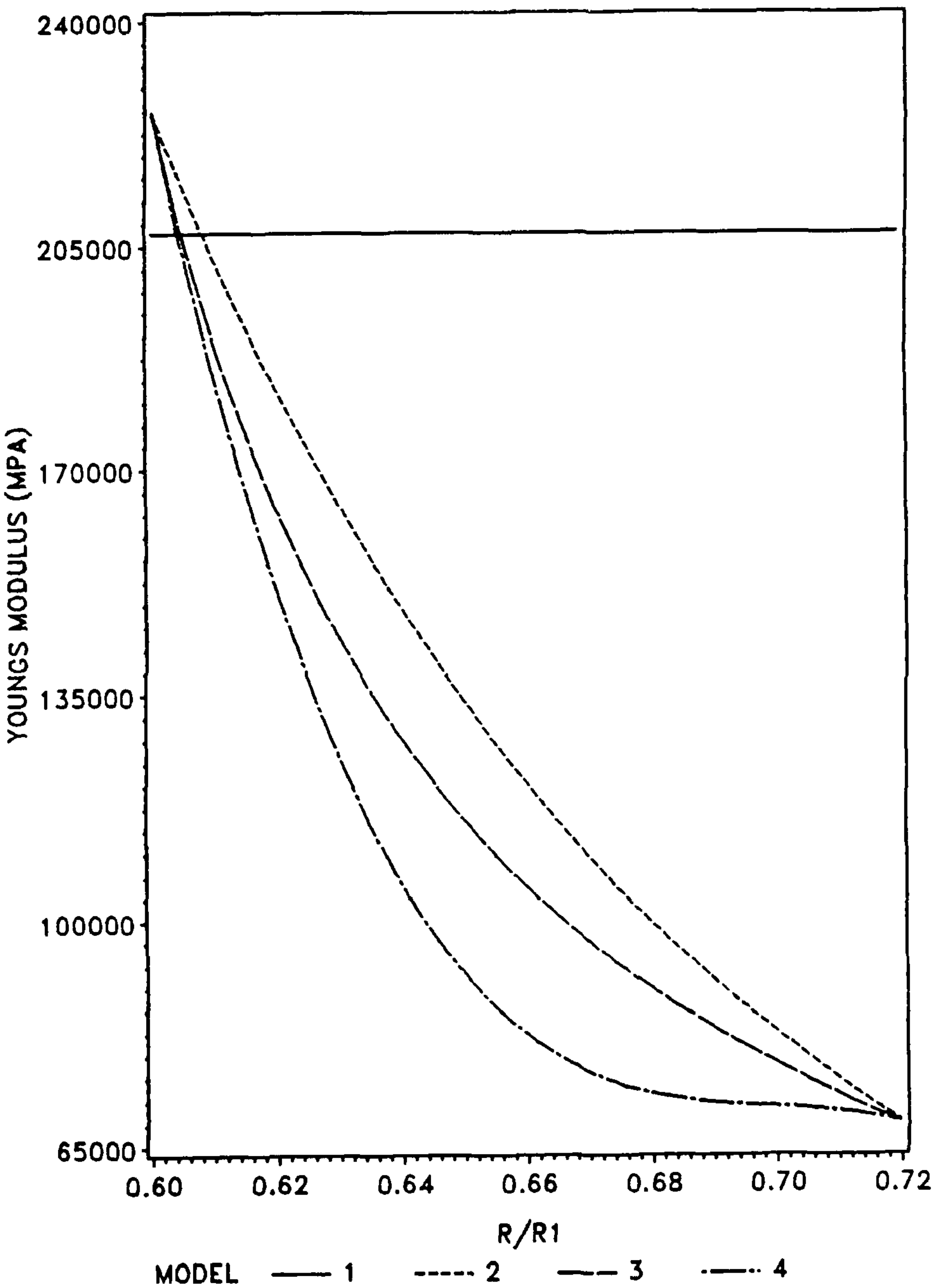

Figure 13. Plot of four material variation models used to represent the material modulus in the interphase.

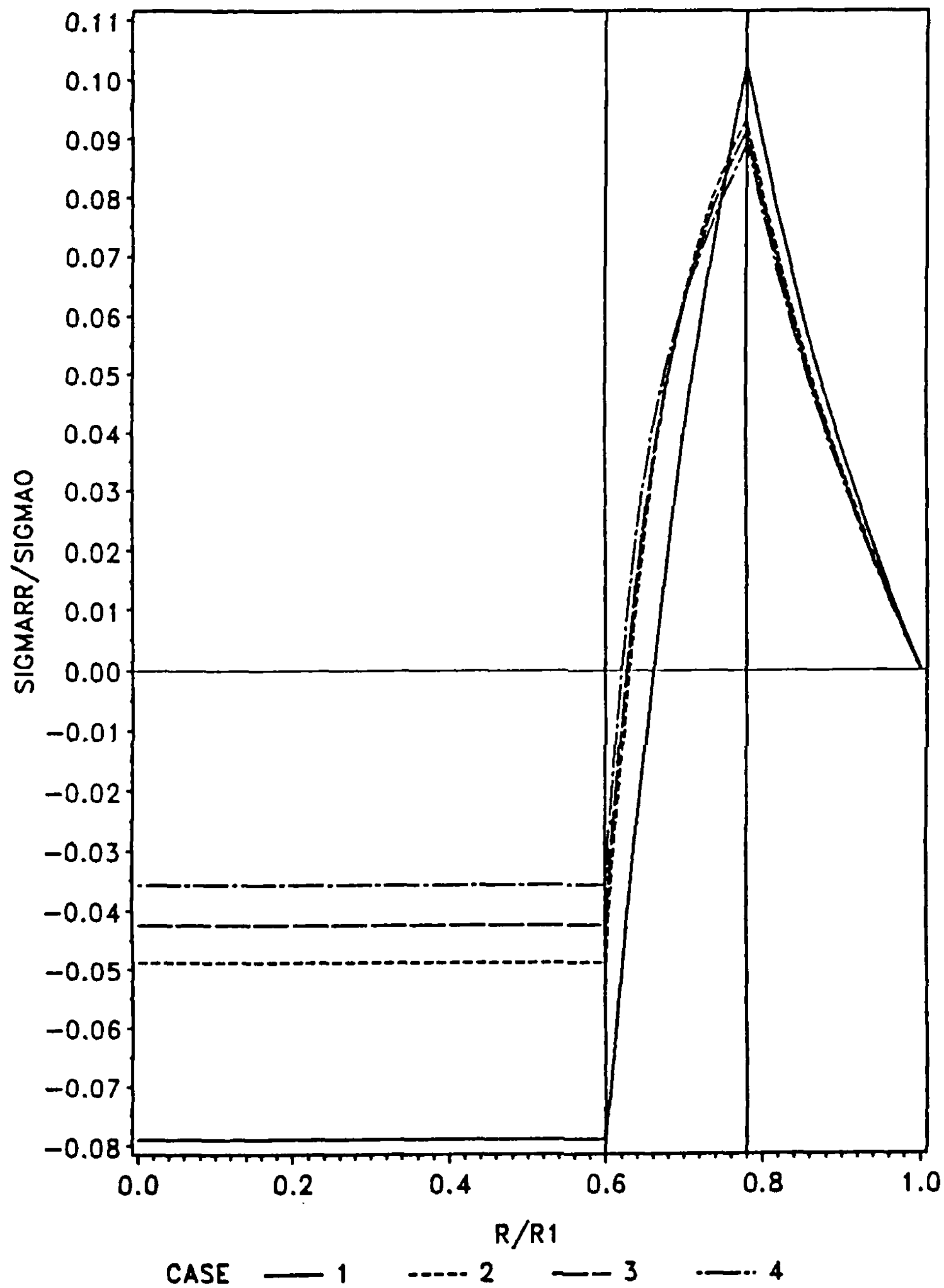

Figure 14. Plot of radial normal stress in the fiber-interphase-matrix assembly for the four cases considered.

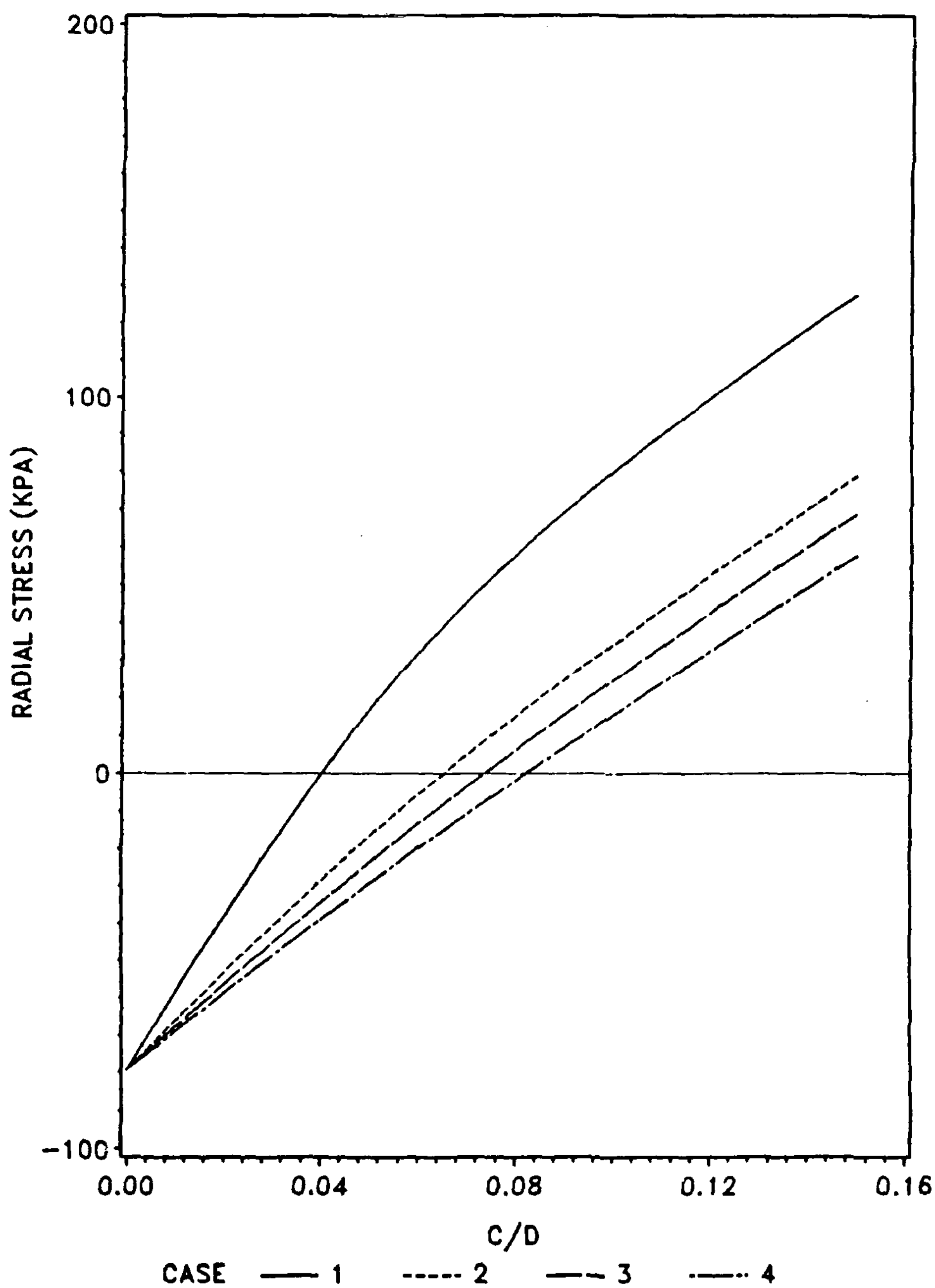

Figure 15. Plot of radial normal stress as a function of the thickness of the interphase region divided by the fiber radius for the four cases considered.

- A reciprocal variation of the form $\frac{P}{(r - Q)}$
- A cubic variation of the form $Pr^3 + Qr^2 + Rr + S$

where P, Q, R, and S are constants.

For demonstration purposes, the assembly was subjected to a uniform temperature change, and the micro-stresses were determined. The nature of the variations of the elastic modulus is shown in Figure 13. The solid curve for "model 1" corresponds to the classical assumption that the properties of the material in the interface are constant. Curves 2, 3, and 4 correspond to the variation forms mentioned above.

An example of the influence of the nonconstant material properties is shown in Figure 14which is a plot of the normal stress in the radial direction as a function of the radial position for the Ni/Al system. This stress component controls fiber-matrix debonding which is critical to the strength and toughness of the composite system. The differences shown are remarkable; variations of 50 percent are found in the value of the interface stress at the fiber boundary for this very realistic case.

Another illustration of the importance of this approach is shown in Figure 15. In that figure the same radial normal stress is shown as a function of the thickness of the interphase region, for the four different variations of the material properties in that region as discussed above. The normal stress can be varied from tension to compression just by changing the thickness of the interphase region, a most remarkable result. Moreover, the correct thickness of the interphase region cannot be determined unless a correct representation of the properties and their variation in that region is used, as can be seen from the figure. This is an excellent illustration of the importance of the present approach to this problem.

This work continues, with efforts to match the representation with experimental data for the the variations in the interphase. Such data are difficult to find. Further work also continues to examine the influence of nonuniform interphase properties on the micro-stresses under various types of mechanical loading. A more complete presentation of these and other results will be made in a subsequent report.